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Work performed for
U.S. DEPARTMENT OF ENERGY
Nuclear Energy
Reactor Systems Development and Technology
Washington, D.C. 20545
Under Interagency Agreement DE-AL03-86SF16310

Prepared for
Fourth Symposium on Space Nuclear Power Systems
sponsored by the University of New Mexico
Albuquerque, New Mexico, January 12-16, 1987

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SUMMARY

The high vacuum creep and creep-rupture behavior of a Nb-1Zr-.1C alloy (PWC-11) was investigated at 1350 and 1400 K with an applied stress of 40 MPa. The material was tested in the following four conditions: annealed (1 hr 1755 K/2 hr 1475 K); annealed plus EB welded; annealed plus aged for 1000 hr at 1350 or 1400 K; and annealed, welded, and aged. It was found that the material in the annealed state was the most creep-resistant condition tested, and that aging the alloy for 1000 hr without an applied stress greatly reduced that strength; however, it was still approximately three times as creep resistant as Nb-1Zr. Additionally, the EB weld region was stronger than the base metal in each condition tested, and phase extraction of the dispersed precipitate revealed the presence of a 70%ZrC-30%NbC cubic monocarbide phase.

INTRODUCTION

Advanced space power systems to produce electrical power at levels ranging from several kilowatts to many megawatts will require advanced materials to meet stringent mass and performance requirements. These requirements, which may include a service life of greater than 7 yr at a temperature in excess of 1350 K, dictate the use of refractory metals (Cooper 1984). The Nb-1Zr alloy has been suggested for previous applications (Lane and Ault 1965, Buckman 1984), but was not developed for applications which require high creep strength at temperatures in excess of 1100 K.

A Nb-1Zr-.1C alloy, PWC-11, was developed in the early 1960's by the Pratt and Whitney Aircraft Corporation (DeGrosso et al. 1967) for elevated temperature service. Through experimentation with carbon levels and various thermo mechanical processing treatments, it was shown that the tensile and creep strength may be about three to four times that of the base Nb-1Zr composition. Overaging of this alloy in the 1175 to 1375 K temperature range has been of concern because of potential weakening effects (Pratt and Whitney Aircraft 1965).

The study reported herein was conducted to verify previous results and establish the suitability of the PWC-11 alloy as an alternative to Nb-1Zr in space power systems. The following issues were investigated: (1) long-time high vacuum creep behavior, (2) the effect of prior stress-free thermal aging on creep behavior, (3) the effect of electron beam (EB) welding on creep behavior, and (4) the effect of heat treatment and creep testing on the carbide morphology.

MATERIALS AND PROCEDURE

The PWC-11 alloy material used in this study was received from the Oak Ridge National Laboratory in the form of 1 mm thick machined tensile creep specimens. The material contained 0.90 wt % Zr and 0.063 wt % C. Unfortunately, a detailed processing history of this heat is not available. Prior to evaluation or welding, all material was annealed in high-vacuum for 1 hr at 1755 K plus 2 hr at 1475 K as per the manufacturer's recommendations (DeGrosso et al. 1967). Following the anneal, the creep and creep-rupture specimens were subjected to one or both of the following treatments: (1) aging for 1000 hr at 1350 or 1400 K in high vacuum, and (2) single pass full penetration EB welding across the width of the test specimen perpendicular to the tensile axis. This weld orientation causes the stress during testing to be imposed equally on the base metal, weld metal, and heat-affected zone. The welds were surface ground to remove excess material before testing.

RESULTS

A typical microstructure of an EB welded test specimen is shown in figure 1. The base metal away from the weld was in the annealed condition with an average grain size of 25 μm measured by the circle-intercept method, with an aspect ratio of approximately 5:1. The weldment had a columnar structure with the grain size ranging from about 45 μm up to over 200 μm . The annealed condition had an extensive amount of second phase precipitate in the grain boundaries and matrix. The weld zone also showed extensive second phase precipitation; however, the particles appeared to be finer and form cell-like domains within the grain.

Creep tests were conducted in high vacuum (10^{-7} Pa) at 1350 K and 40 MPa to assess the effect of EB welding and aging on creep strength. The creep curves to approximately 1 percent strain (fig. 2) show that the annealed condition required about 3200 hr to achieve 1 percent strain, clearly the most creep resistant state. A similarly treated sample with an EB weldment required 2200 hr, about a 30 percent decrease in the time to reach 1 percent strain. Aged samples in both the welded and unwelded conditions took only about 350 hr to reach 1 percent strain. This approximate order of magnitude loss in the time to reach 1 percent strain is clearly due to the effects of the isothermal stress-free aging treatment.

Moore et al. (1986) conducted short-time creep rupture tests to further characterize the effect of EB welding on the PWC-11 alloy. The EB welds in these tests also were perpendicular to the test axis. Tests were conducted in a 10^{-5} Pa vacuum at 1350 K after the post-weld heat treatment (1 hr at 1475 K) and after the aging treatment (1000 hr at 1350 K). Unwelded specimens with similar heat treatments were tested for comparison purposes. The results of the creep-rupture tests (fig. 3) show that the unaged condition is stronger than the aged condition for both the welded and unwelded specimens. In all creep rupture tests of specimens with a transverse EB weld, failure occurred in the unaffected base metal (fig. 4) thus demonstrating that the weld region was stronger. The upper curve in figure 3, therefore, represents the 1350 K rupture strength of PWC-11 base metal in the annealed condition and the lower curve represents the rupture strength of PWC-11 material annealed then aged for 1000 hr at 1350 K prior to the application of a strain.

The differences in creep and rupture strength noted for PWC-11 material may be due in part to the changes in the precipitate composition and/or morphology. Results of metallographic analysis of several samples annealed and aged with and without an applied stress revealed large metastable carbides in addition to a regular distribution of the stable monocarbide, (Zr,Nb)C. Chemical analysis of these extracted particles showed the composition to be approximately 70 percent ZrC and 30 percent NbC, which correlated well with x ray analysis which showed a cubic phase with a lattice parameter of 0.462 nm. Scanning and transmission electron microscopy revealed the presence of two types of particles presumed to be: (1) very large (5 to 10 μm) particles of metastable Nb₂C, and, (2) oriented (Zr,Nb)C particles, ranging in size from 0.1 to 0.5 μm , some possibly transformed to ZrO₂ during internal oxidation (Korotayev et al. 1980). Grobstein et al. (1986) suggested that isothermal aging without an applied stress caused precipitation and growth of the particles to a size greater than about 0.25 μm , whereas aging in the presence of a stress, such as a creep test, produced a more finely dispersed phase, resulting in more efficient strengthening.

Multiple linear regression analysis of the reciprocal of the time in hours to achieve 1 percent strain was performed on the high vacuum (10^{-7} Pa) creep data (table I) on the PWC-11 alloy. The Dorn parameter (Orr et al. 1954) was determined and the results are shown in figure 5. Extrapolation of the data to 7 yr yielded a projected stress of 20 MPa to limit the creep strain to 1 percent at 1350 K for the alloy in the annealed condition. Extrapolation of the creep strength data of the PWC-11 alloy in the aged condition shows that a stress of about 13 MPa could be applied for 7 yr at 1350 K before 1 percent creep strain would be reached. Although the creep data and extrapolated 7 yr creep strength values show that the PWC-11 alloy may lose approximately 30 percent of its creep strength due to aging at 1350 K, even in this weakened condition it has been shown to be about three times as strong as the Nb 1Zr alloy (Titran 1986).

SUMMARY OF RESULTS

Based upon the preliminary studies of the creep and creep-rupture behavior of the PWC-11 alloy at 1350 and 1400 K, the following observations can be drawn:

- (1) The PWC-11 material in the annealed state (1 hr at 1755 K plus 2 hr at 1475 K) was the most creep resistant condition tested.
- (2) Aging the PWC-11 alloy for 1000 hr at either 1350 or 1400 K without an applied stress greatly reduced the strength of the annealed PWC-11 material; however, it was still more creep resistant than the Nb 1Zr alloy.
- (3) The electron-beam weld region was stronger than the base metal for specimens creep-rupture tested in both the post-weld heat treated (1 hr at 1475 K) and aged (1000 hr at 1350 K) conditions.
- (4) The 70%ZrC-30%NbC cubic monocarbide was the only extracted phase identified by x-ray diffraction and by chemical analysis.

CONCLUSION

Stress-free thermal aging of the PWC-11 material at 1350 or 1400 K prior to the application of stress is detrimental to its creep strength and should be avoided in potential space power systems.

ACKNOWLEDGMENTS

This research was performed by the NASA Lewis Research Center for the U.S. DOE Office of Nuclear Energy and the Strategic Defense Initiative Office under interagency agreement DE-AI03-86SF16310.

REFERENCES

1. Buckman, R.W. Jr.: A Review of Tantalum and Niobium Alloy Production. Refractory Alloy Technology for Space Nuclear Power Applications, R.H. Cooper and E.E. Hoffman, eds., CONF-8308130, DOE, 1984, pp. 86-91.
2. Cooper, R.H. Jr.: Potential Refractory Alloy Requirements for Space Nuclear Power Applications. Refractory Alloy Technology for Space Nuclear Power Applications, R.M. Cooper and E.E. Hoffman, eds., CONF-8308130, DOE, 1984, pp. 14-17.
3. DelGrosso, E.J.; Carlson, E.C.; and Kaminsky, J.J.: Development of Niobium-Zirconium-Carbon Alloys. J. Less Common Metals, vol. 12, 1967, pp. 173-201.
4. Grobstein, T.L.; and Titran, R.H.: Characterization of Strengthening Precipitates in a Nb-Zr-C Alloy. Presented at the TMS-AIME Meeting in Orlando, FL, Oct. 5-9, 1986.
5. Korotayev, A.D. et al.: Influence of the Structural State on the Mechanism by Which a Non-Metallic Phase is Formed During Diffusion Saturation of Niobium Alloys with Oxygen. Phys. Met. Metallogr. (Engl. Transl.), vol. 50, no. 3, 1980, pp. 92-99.
6. Lane, J.R.; and Ault, G.M.: The Refractory Metals Sheet Rolling Program. Met. Eng. Q., vol 5, no. 3, Aug. 1965, pp. 23-29.
7. Moore, T.J.; Titran, R.H.; and Grobstein, T.L.: The Effect of Electron Beam Welding on the Creep-Rupture Properties of a Nb-Zr-C Alloy. NASA TM-88892, 1986.
8. Orr, R.L.; Sherby, O.D.; and Dorn, J.E.: Correlations of Rupture Data for Metals at Elevated Temperatures. Trans. ASM, vol 46, 1954, pp. 113-128.
9. Advanced Materials Program for November and December 1964. PWAC-1018, Pratt and Whitney Aircraft, Middletown, CT, 1965.
10. Titran, R.H.: Long-Time Creep Behavior of the Nb-1Zr Alloy Containing Carbon. Presented at the TMS-AIME Meeting in Orlando, FL, Oct. 5-9, 1986.

TABLE I. - CREEP DATA FOR THE PWC-11 MATERIAL
TESTED IN A VACUUM OF 10^{-7} Pa

Material condition	Test temperature, K	Test stress, MPa	Time to 1 percent strain, hr
A ^a	1350	34.5	3200
A	1350	40.0	3450
A	1400	40.0	355
A	1400	35.0	900
A	1350	45.0	980
A	1400	27.6	2760
A	1350	10.0	(b)
A + 1000 hr/1400 K	1400	35.0	150
A + 1000 hr/1400 K	1350	40.0	168
A + 1000 hr/1350 K	1350	40.0	314
A + 1000 hr/1350 K	1350	10.0	(c)

^aThe A condition is the recommended anneal of 1 hr/1755 K + 2 hr/1475 K.

^bTest in progress; 9300 hr with 0.02 percent total strain.

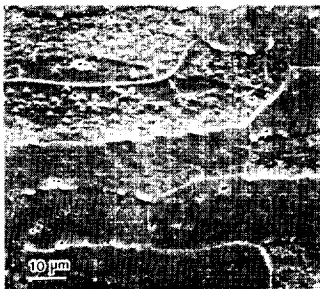
^cTest in progress; 7000 hr with 0.04 percent total strain.

FACE

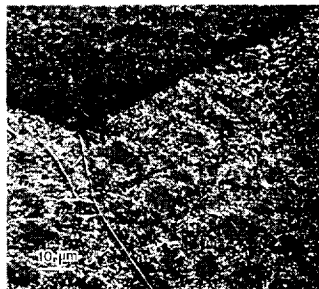


ROOT

(A) CROSS-SECTION OF SINGLE-PASS FULL PENETRATION WELD.



(B) ANNEALED BASE METAL.



(C) WELD FUSION ZONE.

FIGURE 1. - TRANSVERSE ELECTRON BEAM WELDMENT IN PWC-11 MATERIAL IN THE AS-WELDED CONDITION PRIOR TO SURFACE GRINDING AND CREEP TESTING.

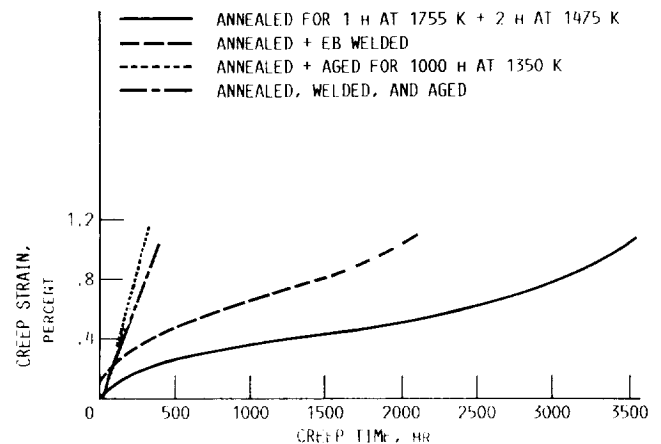


FIGURE 2. - CREEP CURVES FOR PWC-11 MATERIAL WITH AND WITHOUT A TRANSVERSE EB WELD SHOWING THE EFFECT OF A 1000-HR AGING TREATMENT AT 1350 K ON THE TIME TO REACH 1 PERCENT CREEP STRAIN. SAMPLES WERE TESTED AT 1350 K WITH AN APPLIED STRESS OF 40 MPa.

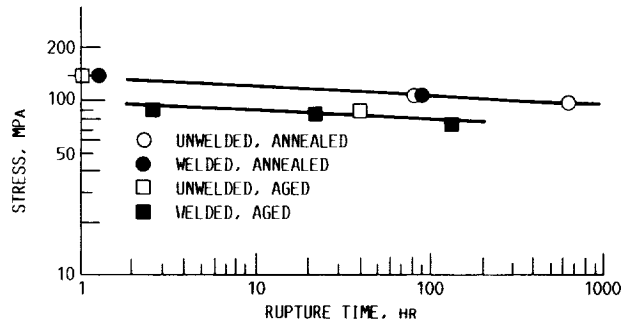


FIGURE 3. - RUPTURE TIME VERSUS APPLIED STRESS AT 1350 K FOR WELDED AND UNWELDED PWC-11 MATERIAL WITH AND WITHOUT A 1000-HR AGING TREATMENT AT 1350 K PRIOR TO TESTING.

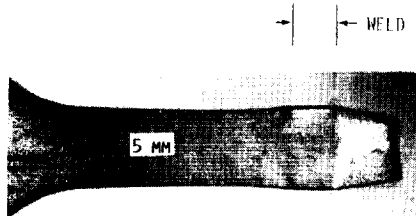


FIGURE 4. - PHOTOMICROGRAPH OF A TYPICAL EB WELDED PWC-11 ALLOY SPECIMEN AFTER CREEP-RUPTURE TESTING SHOWING THAT FAILURE OCCURRED IN THE BASE METAL MATERIAL.

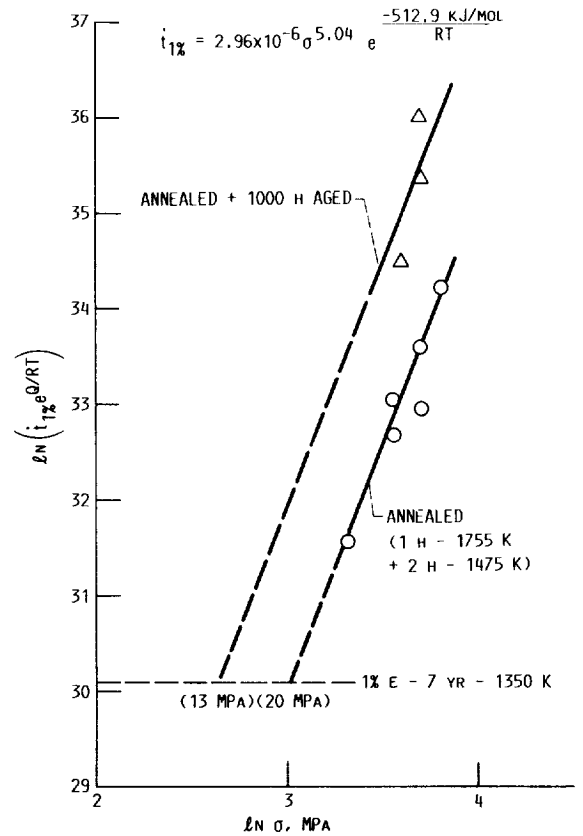


FIGURE 5. - APPLIED STRESS VERSUS THE TEMPERATURE-COMPENSATED TIME TO 1 PERCENT CREEP STRAIN RATE FOR PWC-11 MATERIAL IN THE ANNEALED AND THE ANNEALED PLUS AGED CONDITIONS.

1. Report No. NASA TM-89834		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle Creep Behavior of Niobium Alloy PWC-11				5. Report Date	
				6. Performing Organization Code 506-41-3B	
7. Author(s) R.H. Titran, T.J. Moore, and T.L. Grobstein				8. Performing Organization Report No. E-3306	
				10. Work Unit No.	
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135				11. Contract or Grant No.	
				13. Type of Report and Period Covered Technical Memorandum	
12. Sponsoring Agency Name and Address U.S. Department of Energy Reactor Systems Development and and Technology Washington, D.C. 20545				14. Sponsoring Agency Code Report No. DOE/NASA/16310-3	
15. Supplementary Notes Final Report. Prepared under Interagency Agreement DE-AI03-86SF16310. Prepared for the Fourth Symposium on Space Nuclear Power Systems, sponsored by the University of New Mexico, Albuquerque, New Mexico, January 12-16, 1987.					
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17. Key Words (Suggested by Author(s)) Creep; Nb-base-alloys; EB welding; PWC-11; Carbide precipitation				18. Distribution Statement Unclassified - unlimited STAR Category 26 DOE Category UC-25	
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of pages 7	
				22. Price* A02	